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A method for studying the electrical capacity of a ZrO2-based nanopowder system

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ORIGINAL STUDY

A Method for Studying the Electrical Capacity of a ZrO_2-based Nanopowder System

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Abstract

This article discusses the methodological aspects of the electrical capacitance study of the zirconium dioxide (ZrO_2) based nanoparticles. The methods of selection of the nanoparticle material and the method of its applying are described, along with the technological processes for obtaining dense compacts using high hydrostatic pressure, and the method of its applying are described. A detailed description of the nanoparticle selection, along with the high hydrostatic pressure method used for transforming into dense compacts is provided. The experimental setup, as well, as the technique of electrical contact's application for measuring the electrical capacitance of nanopowder compacts and the results of test experiments are presented.

Keywords: Nanopowders, Zirconium dioxide, Dense compactats, Supercapacitor, Hydrostatic pressure, Impedance spectra

1. Introduction

The transition from fossil energy sources such as oil and coal to renewable sources, including solar, wind and hydropower, is a necessary step to create a sustainable and environmentally safe energy system [1—3]. One of the main advantages of renewable energy is environmental safety, however, despite all its advantages, modern renewable energy sources still have their disadvantages, which must be taken into account when developing energy strategies and making decisions regarding their use [1,4,5].

According to experts [6], the development of modern solid-state electronics is hampered by the lack of submicroscopic capacitors with high capacitance density (>100 μF/g) capable of operating in temperatures above 300 °C and frequencies up to 10 GHz. To date, the basic concept of creating such devices in the form of solid-state nanoion pulse drives of planar design has been defined. They are based on the effect of charge localization at the interface of materials with different types of conductivity (electronic and ionic).

The use of polarizing solid-state materials, semiconductors and dielectrics [7], in particular, solid solutions based on ZrO_2 [8], is promising as working media for such devices. Changing the mechanisms of charge accumulation, namely, transferring the field localization area from the surface (as is the case in carbon supercapacitor) to the volume of the dielectric (creating surface-type structures in the volume of 2D/3D structures) will significantly increase the energy density and electrical capacity of these systems. Such systems have unique physical properties due to the determining role of the phase interface (2D component) in the formation of their electronic properties.

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The aim of the work is to develop a methodology for obtaining and studying the capacitive properties of functional media for solid-state nanoion electric energy storage devices of ultrahigh capacity density.

ZrO_2+3mol%Y_2O_3, pre-annealed at 400—1000 °C.

In this work, the annealing range of 400—1000 °C is used, which is selected taking into account the following factors:

- Fire resistance and structural changes in ZRO_2
  Zirconium dioxide is a high-temperature material in which structural changes and phase transitions occur at relatively high temperatures:
  At temperatures below ~400 °C, there are no significant changes in the structure, the material retains the metastable phases obtained during synthesis.
  At temperatures of 400—500 °C, small crystallites recrystallize and defects decrease, which has a positive effect on conductivity by increasing the packing density.
  At temperatures of 700—1000 °C, phase stabilization (tetragonal and/or cubic) begins due to the diffusion of oxygen vacancies and the redistribution of impurities, as well as the formation of larger grains, which reduces intergranular barriers.
- The effect of temperature on the concentration of oxygen vacancies
  Two important processes are observed during annealing in the range of 400—1000 °C:
  An increase in the concentration of oxygen vacancies due to thermally activated processes. This improves ionic conductivity, as vacancies play the role of the main charge carriers.
  Aggregation of vacancies at higher temperatures (>1000 °C): at too high temperatures, vacancies can aggregate and form complexes, which reduces their mobility and, consequently, conductivity.
- Polymorphic transitions
  In pure ZRO, transitions between phases occur at high temperatures:
  Monoclinic → Tetragonal: begins around 1170 °C.
  Tetragonal → Cubic: above 2370 °C.
  The stabilizing additive Y_2o_3 shifts these temperatures downwards, therefore, stable tetragonal and/or cubic phases can be effectively obtained during annealing in the range of 400—1000 °C. These phases have higher conductivity compared to the monoclinic phase.
- Relationship with morphology and grain size
  The annealing temperature affects the grain size and packing density:
  400—500 °C: nanocrystals with a size of ~8—10 nm are formed, which increases the specific surface area and contributes to the accumulation of charge.
  700—1000 °C: grain increase to ~15—17 nm, which improves density and reduces the number of grain boundaries that interfere with ionic conductivity.
  Temperatures above 1000 °C lead to excessive grain growth, deterioration of the nanostructured nature of the material and a decrease in the density of oxygen vacancies.

Criteria for the selection of YSZ materials for nanoion type capacitors. The size of the electrolyte's structural elements. According to earlier works [9], decrease in the particle size, increases the concentration of free charge carriers in the near-surface layer, as does the amount of free surface energy. Therefore, to intensify the charge transfer processes at the nanoparticle atmosphere surface heterojunction, it is advisable to choose the minimum possible crystallite size of a solid solution based on zirconium dioxide - about 8—9 nm, corresponding to the size of crystalline nanoparticles obtained by heat treatment of hydroxide in the 400 °C, 2 h mode. The minimum particle size is limited by the impossibility of obtaining a dense compact, and, consequently, high volumetric conductivity (the optimal powder for compaction is 700 °C, 2 h, 15—17 nm), therefore, the optimal size of crystallites theoretically lies in the size range 8—17 nm.

Electrolyte material. Choosing the c-ZrO_2 or t-ZrO_2 phase is motivated by the relative simplicity of their structure and the fact that these systems are widely used in many applications.

According to the literature data [10], as the concentration of the alloying impurity Y_2O_3 increases in a solid solution of ZrO_2 - Y_2O_3, the concentration of vacancies capable of participating in charge transfer processes increases, while their mobility decreases as a result of aggregation into complexes (impurity-vacancy complexes). The optimal composition is ZrO_2 + (3—16) % mol Y_2O_3 [11]. Taking into account the dimensional features of charge transfer processes in dispersed nanopowder systems (conductivity decreases as grain size decreases), the composition of the solid solution requires experimental optimization. The choice of a stabilizing impurity concentration of 3 mol.% Yo_3 is due to several key factors related to optimizing the properties of zirconium dioxide to achieve the best conductive characteristics and prevent undesirable phase transitions:

- Inhibition of polymorphism processes
  Zirconium dioxide (ZrO_2) without stabilization has several phases: monoclinic (m), tetragonal (t) and cubic (c). At temperatures below 1000 °C, the monoclinic phase is thermodynamically stable, but it has low ionic conductivity.

The addition of Y_2O_3 helps to stabilize the tetragonal (t) and cubic (c) phases, which have higher conductivity. The concentration of 3 mol.% y_2o_3 is the minimum necessary to stabilize the tetragonal phase at room temperature, which prevents transitions between phases that can destroy the structure of the material.

- Optimization of ionic conductivity

When Yo_3 is added, oxygen vacancies are created, which are necessary for charge transfer. However, an increase in the concentration of the stabilizer above 3 mol.% leads to aggregation of vacancies into complexes, which reduces their mobility and, as a result, ionic conductivity.

Concentration of 3 mol.% is a compromise, providing enough vacancies for high conductivity, but preventing their aggregation.

The authors refer to works that explore wider ranges of YO_3 concentrations (for example, from 3 to 16 mol.%). These works indicate that:

At concentrations below 3 mol.% there are not enough oxygen vacancies for effective charge transfer.

At concentrations above 8—10 mol.% there is a significant decrease in conductivity due to a decrease in vacancy mobility.

A change in the concentration of Yo_3 also affects the particle size:

Lower concentrations (<3 mol.%) lead to smaller grain sizes, but complicate the sintering process due to the low packing density.

Higher concentrations (>10 mol.%) increase the grain size, but worsen the density of oxygen vacancies and reduce ionic conductivity.

For this work, the important parameters are:

The size of the crystallites: The study mentions that the optimal particle size is 8—17 nm, which ensures high packing density and ionic conductivity.

Temperature conditions:Annealing at 400—1000 °C is used to optimize the phase composition and particle size.

Phase composition: Mainly tetragonal or cubic phase stabilization is used to maximize conductivity.

Physical modification of the YSZ nanoparticles surface by metal ions. Surface chemical activity plays a crucial role in the formation of macroscopic properties and characteristics of heterogeneous materials based on zirconium dioxide. Niquel ions provide the YSZ surface with high catalytic activity. The Ni/ZrO_2 system has a small discrepancy between the atomic lattice volumes (Ni and ZrO2), which ensures the coordination of their structural, thermal and mechanical properties [12,13]. Other metals, for many reasons, are less consistent with ZrO_2 [14]. Recently, Ni/ZrO2 surface it is considered promising for application in solid-state fuel cells [15,16], where intensive ion and electronic interphase exchange also takes place.

Spatial geometry of the samples. Compaction of YSZ nanoparticles, into compacts makes possible to obtain the necessary degree of atomic interaction and, as a result, ensure the penetration of wave functions of the electron density of nanoparticles into the adsorbent layer and the formation of a charge distribution equivalent in capacity to an atomically thin double electric layer at the heterogroup. It should be noted that in the case of nanopowder systems connected by interparticle electrostatic interaction, there is a heterogeneity in the density of compacts due to significant repulsive mechanical stresses arising from compression of strong nanopowder aggregates [17].

Using high hydrostatic pressure (HHP), makes it is possible to carry out overall uniform compression of a nanopowder. It is placed in an elastic rubber mold along with some liquid (water, various oils, glycerin), ensuring maximum uniformity of the distribution of nanoparticles in the volume of the compact. In this case, the pressure is evenly distributed in all directions. The product is pressed, obtaining a uniform density to an extent that cannot be achieved by just directional pressing along only one axis. The HPP pressure depends on the shape and size of the final product, the required density, but mainly on the properties of the powder.

Electrical contacts. A serious scientific and technological problem is choosing the appropriate method for applying electrical contacts that would not affect the

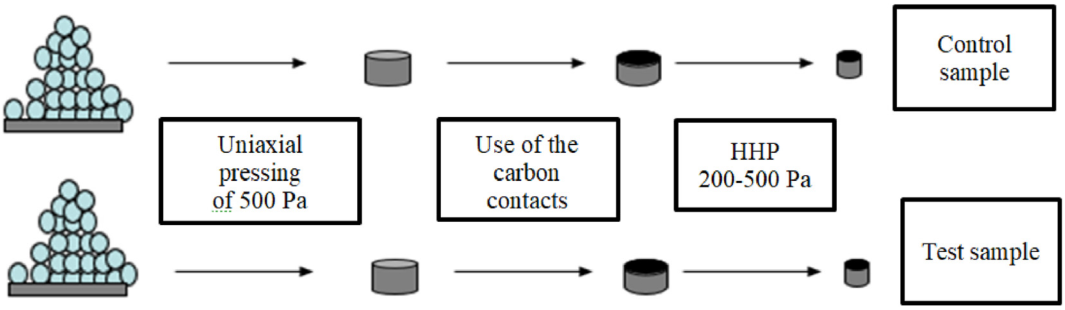

Fig. 1. The sequence of technological operations in the production of YSZ nanoionic capacitors.

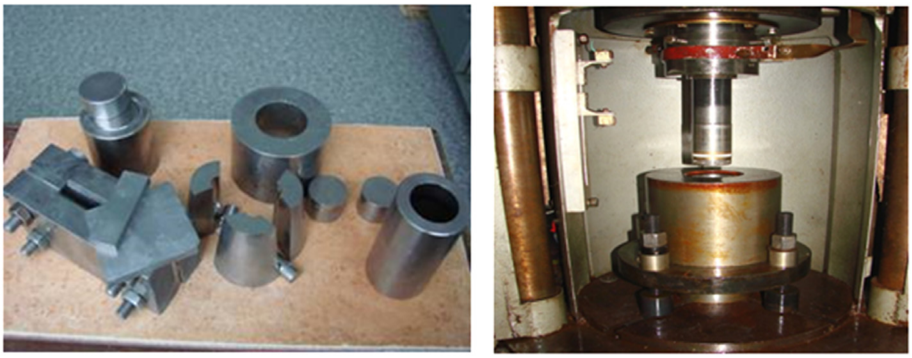

Fig. 2. Moulds for pressing samples and the ATC-2 high-pressure unit.

Fig. 3. The appearance of nanoion capacitors based on YSZ nanopowders.

physical properties of a nanoion capacitor. The contacts obtained by thermal vacuum spraying turned out to be extremely unreliable, since the sprayed metal was poorly retained on the surface of the sample and was very quickly erased. It was previously established [18], that the method of mechanical contact application using a graphite rod, to be the best from a practical point of view.

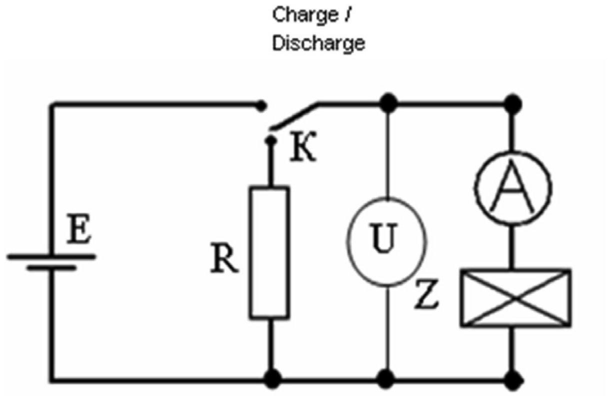

Fig. 5. Electrical diagram for the study of the electrical properties of the sample (Z). R — electrical load, K — switch, E — power supply.

2. Methodology of the experiment

Method for obtaining YSZ based of nanoion capacitor samples. The sequence of technological operations for the production of nanoion capacitors from YSZ nanopowders is schematically shown in Fig. 1.

The powders were first formed by uniaxial pressure into tablets or prism-like objects, and then further compacted by HHP (500 MPa). Pressing was carried

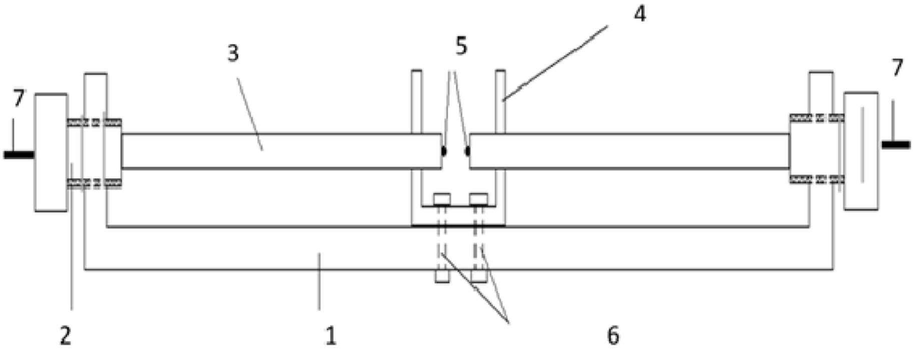

Fig. 4. Schematic representation of a sample holder for studying the electrical capacity of nanopowder compacts, where: 1 - the installation frame; 2 - a spring mechanism that holds ceramic tubes 3 with silver contacts at the ends 5; 4 - a structure that guides and holds the ends of ceramic tubes; 6 - Bolts connecting parts 1 and 4; 7 - wires.

Table 1. Calculated data for the capacity of samples ZrO_2 +3mol%Y_2O_3 with different voltage values measured at humidity η = 50 % and annealing at 400 °C.

V	1 MOhm	10 kOhm	100 kOhm
	μF		
0.5V	13	167.433	29.2502
1V	0.054	446.489	52.5766
3V	0.071	336.653	136.8518
6V	0.0713	751.713	285.4908
10V	0.331	1256.948	727.934

out on presses with a maximum load of 2500 and 65,000 kg. For uniaxial pressing, moulds made of steel were used, in which a sample can be obtained in the form of a cylinder with a diameter of 10, 30 mm and a rectangle of 40 × 60 mm. Hydrostatic treatment of the samples was performed in elastic shells on the UVS-2 press at 100—500 MPa (Fig. 2). Oil was used as the working fluid.

Electrodes were applied on the opposite side of the samples with a graphite pencil. The appearance of nanoion capacitors based on YSZ nanopowders is shown in Fig. 3.

The electrodes of the nanoion capacitor samples were connected to an electric circuit through spring-loaded silver contacts of the sample holder (Fig. 4).

By means of a spring mechanism, ceramic tubes are able to move along the axis of their symmetry, ensuring reliable fixation of the sample in contacts 5.

The schematic diagram of the experimental layout is shown in Fig. 5.

The layout has two modes of operation, which can be conditionally called “charge” and “discharge”. In the “charge” mode, through the switch K (Fig. 5), the electrodes of the sample are connected to the energy source E (250-600 V) for a time until the current in the circuit reaches a stable value (3—6 minutes). In the “discharge” mode, the electrodes of the sample are connected in the same way to an ohmic load R of 1 MOhm. During “charge” stage were measured the electric current through the sample, its voltage, and X-ray diffraction from a surface normal to the electrode surfaces. At “discharge”, a temporary sample's voltage time dependence drops in the temperatures e range of 30—400 °C was registered, as well as, short-circuit. The effect of air humidity on the electrical processes in the samples was investigated. In order to do this, the

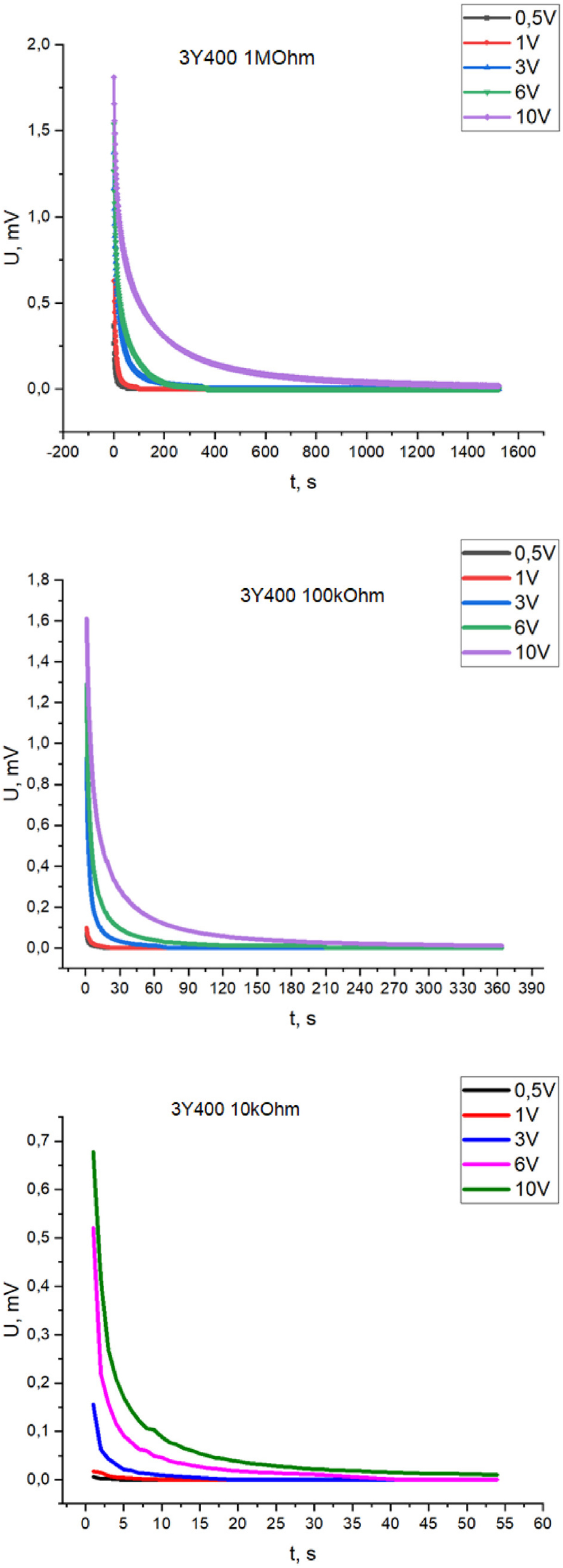

Fig. 6 (a) A group of discharge curves of the initial ZrO_2 +3 mol%Y_2O_3 powder, after annealing at 400° C and resistance of 1 MOhm. (b) A group of discharge curves of the initial ZrO_2 +3 mol%Y_2O_3powder, after annealing at 400 °C and resistance of 10 kOhm. (c) A group of discharge curves of the initial powder ZrO_2 +3 mol%Y_2O_3, after annealing at 400°C and with a resistance of 100 kOhm.

compact was treated with water vapour before "charging" until its weight increased by 1—5%.

The discharge curves were recorded using a portable measuring analyzer with an integrated analog-to-digital MS 8250D converter (MASTECH company). The analyses were carried out at 18°C and atmospheric pressure.

The capacity of the samples at direct current C was determined by the formula:

$$C = t / (R_0 \ln U_0 / U) \tag{1}$$

3. Results and discussion

Fig. 6 shows a family of discharge curves of samples of the ZrO_2 + 3mol% Y_2O_3 system obtained by annealing at 400 °C. It can be seen that the exposure of the samples to an electric field of the order E = 0.5; 1; 3; 6; 10 V/mm for t = 10 seconds, at humidity of η = 50 %, leads to the induction of a potential difference from 1V to 2V on their electrodes.

The maximum values of the residual voltage U_0 on the electrodes are observed at 10V. The minimum value of the residual voltage is observed at 0.5 V. The discharge curves (Figs. 2—4) are exponential and can be extrapolated by the equation:

$$U = U_0 \exp(-t / R_0 C) \tag{2}$$

where U_0 is the value of the voltage on the electrodes during switched off field; R = 1 MOhm is the resistance of the discharge circuit; t_{dis} is the discharge time. U = U_0/e, is the time at which the voltage of the sample decreases by e = 2.718 times.

The capacitance values for the curves in Fig. 1 are shown in Table.1. The capacitance value increases rapidly with the increasing voltage. The maximum capacity C_{max} occurs at a load of 10 kOhm and a voltage of 10 V (Fig. 6b) and amounts to C_{max} = 1256.948 μF. Fig. 6 presents with the curves of the samples. The experiment was conducted at three different values of resistance: 1 MOhm (Fig. 6a), 10 kOhm (Fig. 6b), and 100 kOhm (Fig. 6c).

General conclusions on these curves:

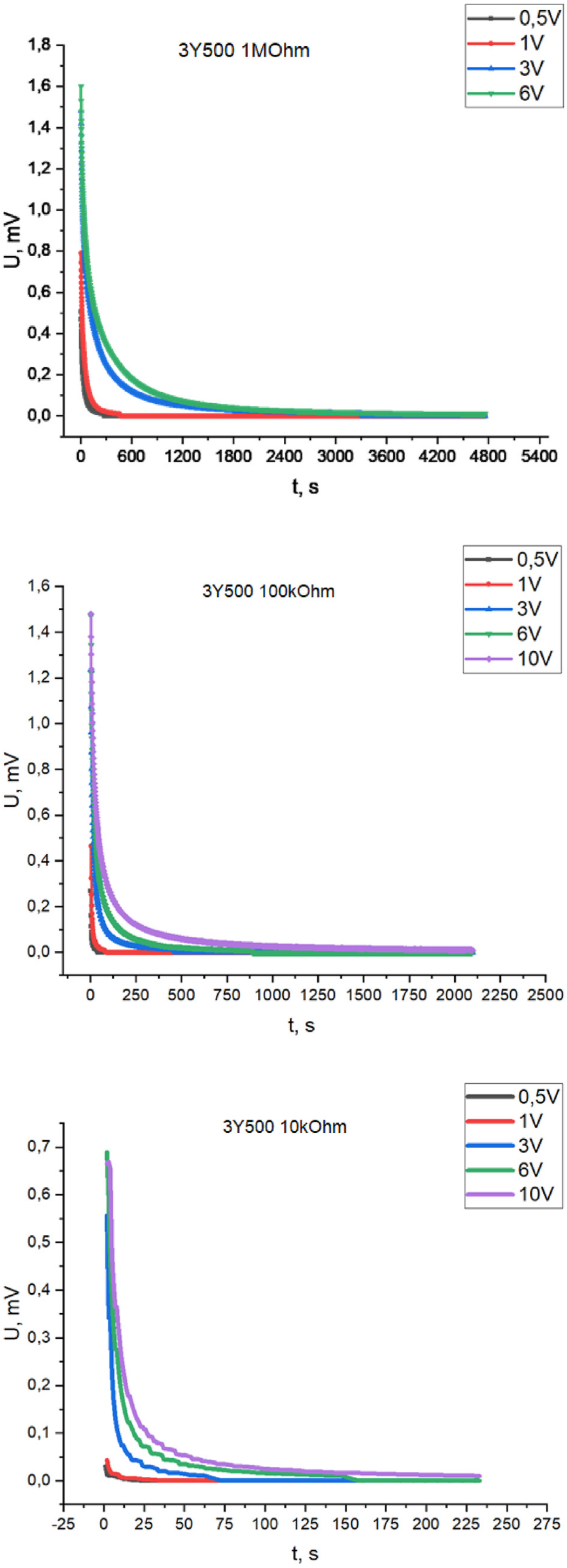

Fig. 7 (a) A group of discharge curvesr of the initial ZrO_2 +3 mol%Y_2O_3 powder, annealed at 500°C, and with a resistance of 1 MOhm. (b) A group of discharge curves of the initial ZrO_2 +3 mol%Y_2O_3 powder, annealed at 500°C and with a resistance of 100 kOhm. (c) A group of discharge curves of the initial ZrO_2 +3 mol%Y_2O_3 powder, annealed at 500 °C and at a resistance of 10 kOhm.

1. With an increase in the applied voltage from 0.5 to 10 V, there is a significant increase in the residual voltage U_0 on the electrodes.
2. The capacitance reaches a maximum at 10 kOhm and 10 V, which is confirmed by the value $C_{max} = 1{,}256{,}948$ μf (Fig. 6b).
3. The discharge curves are exponential and can be described by the equation $U = U_0 exp\ (-t/R0C)$, where R_0 is the resistance of the discharge circuit, and t_{dis} is the discharge time.

Fig. 7 shows the discharge curves for the ZrO_2 +3 mol%Y_2O_3 system obtained by annealing 500 °C. The experiment was carried out at resistances of 1 MOhm (Fig. 7a) and 100 kOhm (Fig. 7b). Highlights:

1. When both samples are compared the annealed at 500 °C displays a general increase in capacitance, which is associated with a structural and density change of the sample.
2. If the resistance values are being compared, with an increase in resistance, a slower discharge is observed, which indicates the dependence of the discharge characteristic on the resistance of the discharge circuit.

An increase in the annealing temperature from 400 °C to 500 °C led to an improvement in the capacitance characteristics of the samples, which can be attributed to changes in the microstructure of the material. The exponential nature of the discharge and the dependence of capacitance on resistance and voltage make it possible to effectively simulate the behaviour of nanoion capacitors and optimize their characteristics for practical use.

In accordance to Table 2 in the case of the ZrO_2 +3 mol%Y_2O_3 system obtained at 500°C, the maximum capacity is reached at a load of 10 kOhm (977 μF/g). Based on this, it can be concluded that at this load, the system is parametrically coherent, i.e., its impedance is in the <10 kOhm region.

Table 2. Calculated data for the capacity of samples ZrO_2 +3mol%Y_2O_3 with different voltage values measured at humidity $\eta = 50$ % and after annealing at 500°C.

3Y500			
V	1 MOhm	10 kOhm	100 kOhm
	μF		
0.5V	38.315	310.7	17.66
1V	50.949	222.1	35.49
3V	118.68	323.385	170.2
6V	158.58124	637.897	316.642
10V	–	976.841	747.651

4. Conclusions

In the course of the work, a methodology was developed for obtaining samples based on zirconium dioxide (ZRO_2) nanoparticles with the addition of 3 mol.% Yo_3 to study their capacitive properties. Thermal annealing of the samples was carried out in the temperature range of 400–1000 °C, which made it possible to stabilize the tetragonal phase, optimize the concentration of oxygen vacancies and improve conductivity.

The experimental data allowed us to determine the key parameters affecting the electrical capacitance and conductive properties of the material.

The reasons for the change in conductivity in zirconium dioxide-based materials are structurally related to several key factors:

Oxygen vacancies play a critical role in the mechanisms of ionic conductivity in zirconium dioxide-based materials, especially in stabilized systems such as $ZRO_2 + Y_2O_3$.

When Y_2o_3 (or other stabilizing additives) are added, some of the ZR^{4+} ions are replaced by $Y3^+$ ions. This leads to a mismatch of charges and the appearance of oxygen vacancies to maintain electrochemical equilibrium. The vacancy concentration is proportional to the amount of stabilizer added.

Oxygen vacancies contribute to ionic conductivity due to the possibility of their movement in the structure of the material. The higher the vacancy concentration, the higher the ionic conductivity. However, at high concentrations of vacancies, they can aggregate, which reduces their mobility.

The thermal effect on the samples changes the distribution of oxygen vacancies, as well as their interaction with other structural defects:

When the temperature rises, vacancies can aggregate or interact with dopants, forming complexes (for example, vacancies-an impurity ion). This reduces their mobility and, as a result, their conductivity.

Thermal annealing also affects the microstructure of the material (grain size and crystallites), which is associated with changes in the density and distribution of vacancies. For example, annealing at higher temperatures can help to improve the ordering of the structure, reducing defects.

In addition to oxygen vacancies, the conductivity is determined by the morphological characteristics of the material:

- As the grain size decreases, the specific surface area increases, which enhances surface effects such as the accumulation of charges at the boundaries. This can both increase and decrease the conductivity, depending on the nature of the grain boundaries.

- Densely packed structures have fewer intergranular barriers, which improves conductivity. At the same time, porous structures can interfere with the effective transfer of ions.
- The phase composition (tetragonal or cubic phases of ZRO_2) also contributes to the conductivity, since different phases have different ion mobility.

The change in conductivity in the $ZRO_2 + Y_2O_3$ system is closely related to the concentration of oxygen vacancies, their interaction under the action of heat and the morphological features of the material. For a deeper understanding of the mechanism, further investigation of the vacancy distribution, particle size, sample density and their phase composition is required.

At a temperature of 400 °C, the formation of nanocrystallites with a size of ~8–10 nm is observed. This ensures a high concentration of oxygen vacancies and a significant specific surface area. However, the conductivity is limited by high intergranular resistance.

Annealing at 500 °C resulted in an increase in capacity due to an optimal combination of fine grains (~12 nm) and increased material density.

At a temperature of 700–1000 °C, grain growth is observed to ~ 15–17 nm and a decrease in structural defects, which minimizes intergranular barriers and increases conductivity. The maximum capacity (977 μF/g) is reached at these temperatures.

With an increase in the resistance of the discharge circuit (up to 100 kOhm), the discharge duration increases, which allows deeper investigation of the charge transfer processes in the material.

An increase in voltage from 0.5 to 10 V showed that the capacitance increases with voltage, reaching a peak at a load of 10 kOhm.

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Conflict of interest

The authors have no conflicts of interest to declare that are relevant to the content of this article.

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